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Centre canadien de la technologie des minéraux et de l'énergie

ISO/TC 102/SC 2 - 15th INTERNATIONAL MEETING
LONDON, UK, 1990-09-24/28

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ISO/TC 102/SC 2 - 15 th INTERNATIONAL MEETING

LONDON, UK, 1990-09-24/28

by

R. Sutarno*

(Convener of ISO/TC 102/SC 2/WG 12B)

This report covers the part of the proceedings of the 15th International Meeting of ISO/TC 102/SC 2 dealing with the work of ISO/TC 102/SC 2/WG 12B (Working Group on Evaluation of Statistical Data, Technical Committee 102 - Iron Ores, Subcommittee 2 - Chemical Analysis). Other topics discussed at the meeting have been reported by the leader of the Canadian delegation.

Keywords: ISO, iron ores, chemical analysis.

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ISO/TC 102/SC 2/WG 12B MEETING

This meeting was called in order to discuss in detail various statistical problems faced by the sub-committee. It was attended by fifteen delegates interested in statistics.

Date: 1990-09-25
Time: 9.00 to 12:00
Place: British Standards Institution Office

Attendance: Nineteen delegates attended.

A. TOPICS DISCUSSED:

(1). Convener's report

The Convener reported the working group activities during the period of 1988 to 1990. Details, annexed to this report, were tabled in the subcommittee meeting as document ISO/TC 102/SC 2 (WG12-90/4).

(2). Liaison with ISO/TC 69/SC 6

The convener reported the activity of ISO/TC 69/SC 6 (document ISO/TC 102/SC 2 (WG12-90/3) N 1017. The delegates agreed that the practice used by the iron ores analytical community in obtaining duplicate determinations does not satisfy the repeatability condition, as defined by ISO/TC 69. For this reason, it was decided that the term repeatability (and its derived terms) used in all ISO/TC 102/SC 2 documents will be replaced by "independent duplicate". Thus the term repeatability limit will be replaced by "independent duplicate limit". The symbol "r" will be replaced by "R_d".

The delegates present discussed the necessity of establishing liaison with ISO/REMCO. It was agreed to recommend to ISO/TC 102/SC 2 to have a formal liaison with ISO/REMCO in order to ensure that the subcommittee is kept informed on the development of reference materials. In addition, the subcommittee agreed to form a new working group (WG 34) to write standards for iron ore certified reference materials. Canada has been requested to provide the convenership.

(3). Common clauses for expression of precision data

As an effort to maintain the uniformity of standards for chemical analyses, a set of common clauses for expression precision data was presented and discussed. The delegates agreed that these clauses as presented in document ISO/TC 102/SC 2 (WG12-90/2), modified to take into account the comments made at the meeting, will be included in all chemical analysis standards published by the subcommittee. WG 12 was requested to submit the draft of these common clauses to the Secretariat as soon as possible.

(4). The overall permissible difference between loading and unloading laboratories

One of the most important uses of ISO standard methods is to provide values for settlement of an iron ore consignment. In many situations, the consignment is sampled and analyzed at both loading and unloading ports using agreed standard methods for sampling, sample preparation and analysis. Because of errors inherent in each stage of this procedure, the results will unlikely be exactly the same. There are two types of error causing this difference; statistical and systematic errors. The objective is to ensure that the standard procedures are applied correctly both by loading and unloading laboratories, i.e. to guard against systematic error being introduced by either one of the laboratories. The overall permissible tolerance is the value below which the absolute difference between the test results obtained by the loading and the unloading laboratories, each using samples taken from their own sampling installation, may be expected to lie with a probability of 95%. This is an important value for contractual purposes. The subcommittee agreed to request the sampling subcommittee to provide this value.

(5). Conformity with the new IEC/ISO directives

The delegates agreed that the method of operation of the subcommittee should be in conformance with the new IEC/ISO directives using project management methods; thus WG 12 is requested to modify ISO/TC 102/TCR 4 and TCR 5 to achieve this objective.

B. SUMMARY AND RECOMMENDATION

One of the difficulties in using ISO standards for chemical analysis of iron ores is to control their accuracy. This is usually achieved by means of application of the standard to a certified reference material (CRM). The problem is that there is no international standard available yet for the preparation and characterization of CRMs. An ISO standard that specifies the method for preparation, characterization and certification of iron ores will be a great help. This task was assigned to the newly formed working group (WG 34) which Canada has been requested to convene.

In trying to respond to the demand from industry for more and better ISO standards in as short time as possible, ISO/TC 102 is trying to improve its efficiency by implementing IEC/ISO directives. WG 12, of which Canada is the convener, has been assigned to ensure the implementation of these directives.

As these ISO standards for chemical analysis of iron ores are essential for the flow of international trade, any attempt to improve their availability should be encouraged. Therefore, support by CANMET and the Standards Council of Canada for the above working groups is strongly recommended.

ISO/TC 102/SC 2 - 15th INTERNATIONAL MEETING

LONDON, UK, 1990-09-24/28

ANNEX A

ISO/TC 102/SC 2/WG 12

ACTIVITY DURING THE PERIOD OF 1988/1990

1. Revised all regression equations for all methods under revision.

   ISO/DP 2598, ISO/DP 4686, ISO/DP 4688, ISO/DP 9516, ISO/DP 9682, ISO/DP 9683, ISO/DP 9684, ISO/DP 9685, ISO/DP 9685, ISO/DP 10123, ISO/DP 10124.

   (Titles of these documents are listed in Document ISO/TC 102/SC 2 N946E and are available on file)

2. Working on general expression for precision data in the standard methods (Doc. 2 N 948 E).

3. Revising ISO/TC 102/TCR 4 - "Procedure for statistical evaluation of analytical data resulting from international test" and TCR 5 - "Working group operating procedure.

4. Continuing to work on accuracy of Fe determination in cooperation with SC 2/SG 4.

5. Two international tests: WG 31 - Tin. WG 31 data is in but WG 30 has just started.

6. Assisting SC 1, SC 3 and SC 5 in statistical evaluation of some of their data.

7. Correspondence:

   (a). Letter from Mt. Newman Mining Co. Re: Modified regression equation for precision vs. level of concentration.

   (b). Letter from Dr. Koch, Germany on the difference between "σ" and "s" for expressing standard deviations.

ISO-IUPAC-AOAC HARMONIZATION

On behalf of this working group, I attended the fifth Harmonization meeting which was held in Washington last April 1989. Unfortunately, in my opinion, no significant progress has been made. The reasons for this failure is the lack of "status" of the attendance. Any one who attend the meeting was given the same status whether he/she represented any organization or not. In my opinion, the proper procedure for harmonization is by meeting between the representatives (those who look after the statistical matters) of each of the organizations concerned. I understand, however that this harmonization effort is still continuing. The next meeting will be in Geneva this spring. In the mean time, there is an effort to harmonized the statistical matters between Technical Committees within ISO.

ISO/TC 102/SC 2 - 15th INTERNATIONAL MEETING

LONDON, UK, 1990-09-24/28

ANNEX B

LIAISON REPORT ON

ISO/TC 69/SC 6 ACTIVITIES FOR THE PERIOD OF OCTOBER 1988 - AUGUST 1990

This report covers the activities of ISO/TC 69/SC 6 during the period of October 1988 to date which are of interest of ISO/TC 102.

1. ISO/TC 69/SC 6 met twice during this period: November 13-17, 1989 in Berlin, Germany and July 23-27, 1990 in Ottawa, Canada.

2. ISO/TC 69/SC 6/WG 1 (Revision of ISO 5725)

ISO 5725 "Accuracy (trueness and precision) of test methods and results" is a generic standard dealing with assessment, uses and control of accuracy of measurement methods and results. As part of its effort towards harmonization, ISO strongly encourages all its technical committees to use ISO 5725 (when published) for their standard development. ISO 5725 is undergoing major revision. The new version was originally planned for publication in 1990; however, due to the extent of the revision, the publication date will be delayed. The standard is divided into six parts. ISO 5725 Part 1, 2, 4 and 6 have been submitted to the Central Secretariat for circulation as DIS and Part 3 will be submitted as DIS soon after having been modified in light of the comments made at the Ottawa meeting. Part 5 is still to be written by a USA member of ISO/TC 69/SC 6.

ISO 5725 - Accuracy (trueness and precision) of measurement and results
Part 1 - General principles and definitions.
Part 2 - A basic method for the determination of repeatability and reproducibility.
Part 3 - Intermediate measures of precision of a test method.
Part 4 - Basic methods for estimating the trueness of a test method.
Part 5 - Alternative methods for the determination of precision of measurements and results.
Part 6 - Practical application.
- Section 1 - General.
- Section 2 - Determination of limits.
- Section 3 - Methods for checking the acceptability of test results and determining the final quoted result.
- Section 4 - Checking method for stability of test results within laboratory.
- Section 5 - The use of repeatability and reproducibility in assessing laboratories.
- Section 6 - Comparison of alternative test methods.

3. ISO/TC 69/SC 6/WG 3 - Measurement uncertainty.

This working group is trying to develop standards on the expression of uncertainty of measurement results. No significant progress can be reported so far.

4. ISO/TC 69/SC 6/WG 4 - Statistical aspects of reference materials.

This is a joint working group between ISO/REMCO and ISO/TC 69/SC 6. This working group was responsible for the development of ISO Guides 33 and 35 and draft ISO 5725 Part 4. The present work is to develop a standard on the use of reference materials for calibration. A document on this subject is currently under development by the USA.

5. ISO/TC 69/SC 6/WG 5 - Limit of determination.

This working group was formed at the Berlin meeting in 1989. It had its first meeting in Ottawa. The purpose of this working group is to develop standards on the statistical aspects of limit of determination/and or detection of measurement methods such as chemical analysis. No significant progress has yet to be reported.

6. Accuracy 91.

In an attempt to foster harmonization among ISO/TC's on their procedure for the assessment, uses and control of accuracy of international standard methods under their responsibility, ISO Central Secretariat in cooperation with ISO/TC 69 and ISO/REMCO is planning to sponsor Accuracy 91, a workshop on protocol for assessment and control of accuracy of international standard methods. Announcement regarding this workshop will be issued by ISO Central Secretariat shortly.

"ACCURACY 91"

A WORKSHOP ON PROTOCOLS FOR ASSESSMENT AND CONTROL OF ACCURACY OF INTERNATIONAL STANDARD METHODS.

Geneva, September 23-24, 1991

Accuracy is one of the most important criteria of an international standard method of measurement (ISO standard). Accuracy of an ISO standard must be assessed during the development and must be controlled during the routine application of the standard. Both the assessment and the control of the precision are carried out by the proper application of statistical methods, while the assessment and the control of the trueness are carried out by proper use of suitable reference materials. ISO/TC 69 has been entrusted by ISO Council with the function of advisor to all ISO technical committees in matters concerning the application of statistical methods in standardization. As for matters related to reference materials, ISO Council has a Committee on reference materials, ISO/REMCO. In order to harmonize the work of technical committees within ISO, a good liaison between these two committees and other technical committees is necessary. The purpose of this workshop is to foster such harmonization.

Sponsors: This workshop is sponsored by ISO Central Secretariat in cooperation with ISO/REMCO and ISO/TC 69.

Intended participants: Representatives of ISO committees and interested organizations having liaison with ISO (IUPAC, AOAC etc.).

Location: ISO Central Secretariat, 1 rue de Varembe, Geneva, Switzerland

For details and registration package contact:

"Accuracy 91"
ISO Central Secretariat
Case postal 56
CH-1211 Geneve 20, Switzerland.
Telefax: + 41 22 733 3430

ISO/TC 102/SC 2 - 15th INTERNATIONAL MEETING

LONDON, UK, 1990-09-24/28

ANNEX C

Determination of Phosphorous Content in Iron Ores by Molybdenum Blue Spectrophotometric Method (WG 28A

EXAMPLE OF CLAUSE FOR NUMBER OF DETERMINATIONS

Number of determinations

Carry out the determination in duplicate, as far as possible under repeatability conditions* on each test sample. Follow chart in Figure (XX) to compute the final result.

* **repeatability conditions:** Conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

EXAMPLE OF CLAUSE FOR PRECISION EXPRESSION

A. Precision expression

The precision of this analytical method is expressed by the following equations:

Repeatability standard deviation: $\sigma_r = 0.0124\ x + 0.0004$

Between-laboratory standard deviation: $\sigma_L = 0.0188\ x + 0.0001$

Repeatability limit: $r = 0.0350\ x + 0.0011$

Permissible tolerance: $P = 0.0587\ x + 0.0006$

where:
x - concentration of element in the sample
σ_r - repeatability standard deviation
σ_L - between laboratories standard deviation
r - repeatability limit
P - permissible tolerance

A.1. Procedure for obtaining the final result

Compute the following quantities from the duplicate results x_1 and x_2 and process according to Figure (XX).

Mean of duplicates: $\bar{x} = (x_1 + x_2)/2$

Repeatability limit: $r = (0.0350\bar{x} + 0.0011)$

A.2. Between-laboratories precision

Between-laboratories precision is used to determine the agreement between the results reported by two laboratories. The assumption is that both laboratories followed the same procedure described in clause A.1.

Compute the following quantities:

Mean of final results: $\mu_{12} = (\mu_1 + \mu_2)/2$

Permissible tolerance: $P = 0.0587\ \mu_{12} + 0.0006$

where:

μ_1 - the final result reported by laboratory 1.
μ_2 - the final result reported by laboratory 2.

If $|\mu_1 - \mu_2| \leq P$, the final results are in agreement.

A.3. Check for trueness

The trueness of the analytical method can be checked by applying it to a reference material (RM) or certified reference material (CRM). The procedure is the same as that described in clauses above. When the precision has been confirmed, the final laboratory result can be compared with the reference or certified value, A_c. There are two possibilities:

$|\mu_c - A_c| \leq C$ - the difference between the reported result and the reference/certified value is statistically insignificant.

$|\mu_c - A_c| > C$ - the difference between the reported result and the reference/certified value is statistically significant.

where:

μ_c - is the final result of the certified reference material,
A_c - is the reference/certified value of the RM/CRM.
C - is a quantity depending on the type of the RM/CRM used.

A.3.1. Type of Certified Reference Materials (CRM)

CRM's used for this purpose should be prepared and certified in accordance with ISO Guide 35.

(a). CRM Certified by Interlaboratory Test Program

$$\sigma_L = 0.0188\ \mu_c + 0.0001$$

$$\sigma_r = 0.0124\ \mu_c + 0.0004$$

$$C = 2\ [\sigma_L^2 + \sigma_r^2/n + V(A_c)]^{1/2}$$

where:

$V\{A_c\}$ - variance of the certified value, A_c.

(b). CRM Certified by One Laboratory*

$$c = 2\ [2\sigma_L^2 + \sigma_r^2/n]^{1/2}$$

* It is recommended that this type of CRM should be avoided unless the particular CRM is known to have unbiased certified value.

ISO/TC 102/SC 2 - 15th INTERNATIONAL MEETING

LONDON, UK, 1990-09-24/28

ANNEX D

Attendance List

Australia	M. J. Munro (Secretary of ISO/TC 102/SC 2) K. Norish B. W. Zieglaar
Brazil	T. M. De Freitas M. V. De Castillo
Canada	J. C. Hole R. Sutarno (Convener)
China	Jing Ai Fang Song Dao Fa Zhang Yu Chun
France	J. L. Brusset
Italy	M. G. Del Monte Tamba
Japan	Y. Matsumura T. Ohtsubo (Secretary of ISO/TC 102) J. Yoshikawa
Netherlands	H. Umans
South Africa	N. Sidiropoulos
Sweden	G. Airijoki
United Kingdom	A. Cunningham